

Progress in the Flowing Liquid-surface Retention Experiment

J.P. Allain

NPL Associates Inc, Champaign, IL

M. Nieto, M.D. Coventry,

M.J. Neumann and D.N. Ruzic

University of Illinois, Urbana-Champaign

*Department of Nuclear, Plasma and Radiological
Engineering*

Plasma-Material Interaction Group 

ALPS/APEX Meeting, Del Mar, CA

April 15-17, 2002

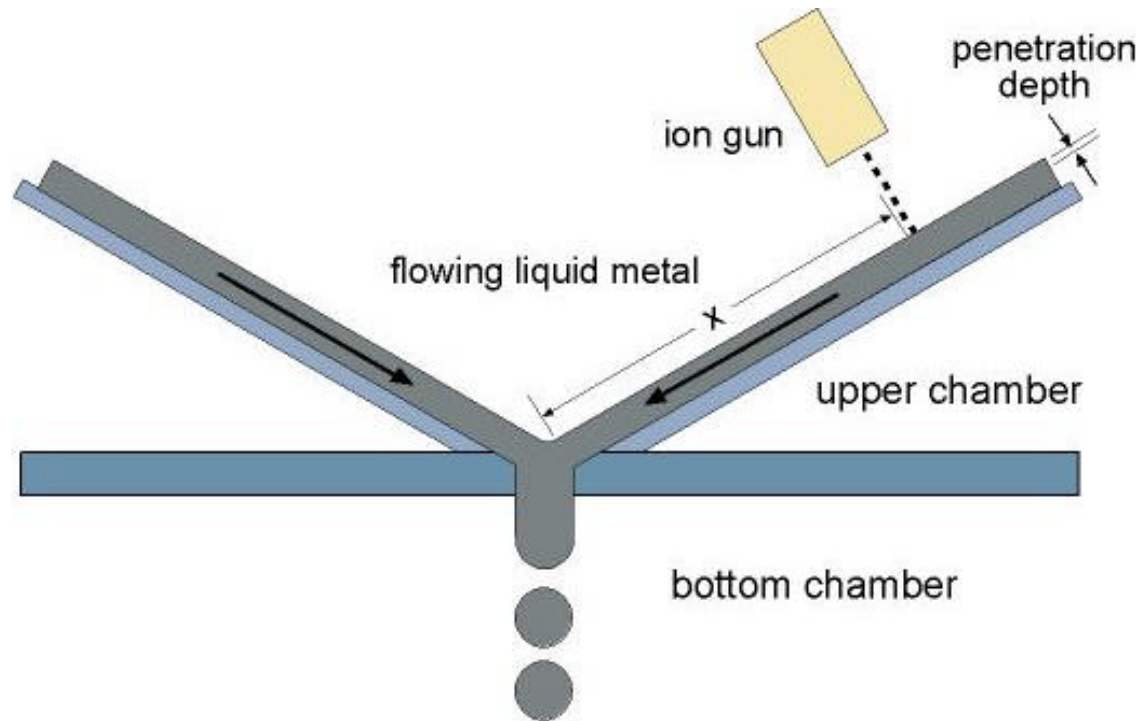
Outline of Talk

- Goals and Objectives of experimental work in FLIRE facility
- FLIRE Experiment
 - Experimental design and setup
 - Lithium loading, melting and transport tests
 - Safety measures in FLIRE
- Liquid lithium injection and flow on internal ramps
- Future plans and upgrades

FLIRE (Flowing Liquid Surface Illinois Retention Experiment) Goals

- FLIRE will provide fundamental data on the retention of He, H, and other gases injected by an ion beam into flowing liquid surfaces such as lithium (Phase I).
- FLIRE will also be upgraded to use a plasma source to generate the incident particles and associated effects (Phase II).
- Further upgrades could modify the experiment to study:
 - high-flux phenomena such as ELMs and disruptions
 - MHD effects on a free surface
 - ability to flow liquid metals through magnetic field gradients
 - effects of eddy-currents

FLIRE concept



- The flowing liquid metal creates a vacuum seal between the upper and bottom chambers.
- Two ramps are provided to ensure the surface layer in which H/He particles are trapped, folds into the middle of the flow and travels to the bottom chamber.
- Flow lasts on the order of 60 seconds, then is recharged

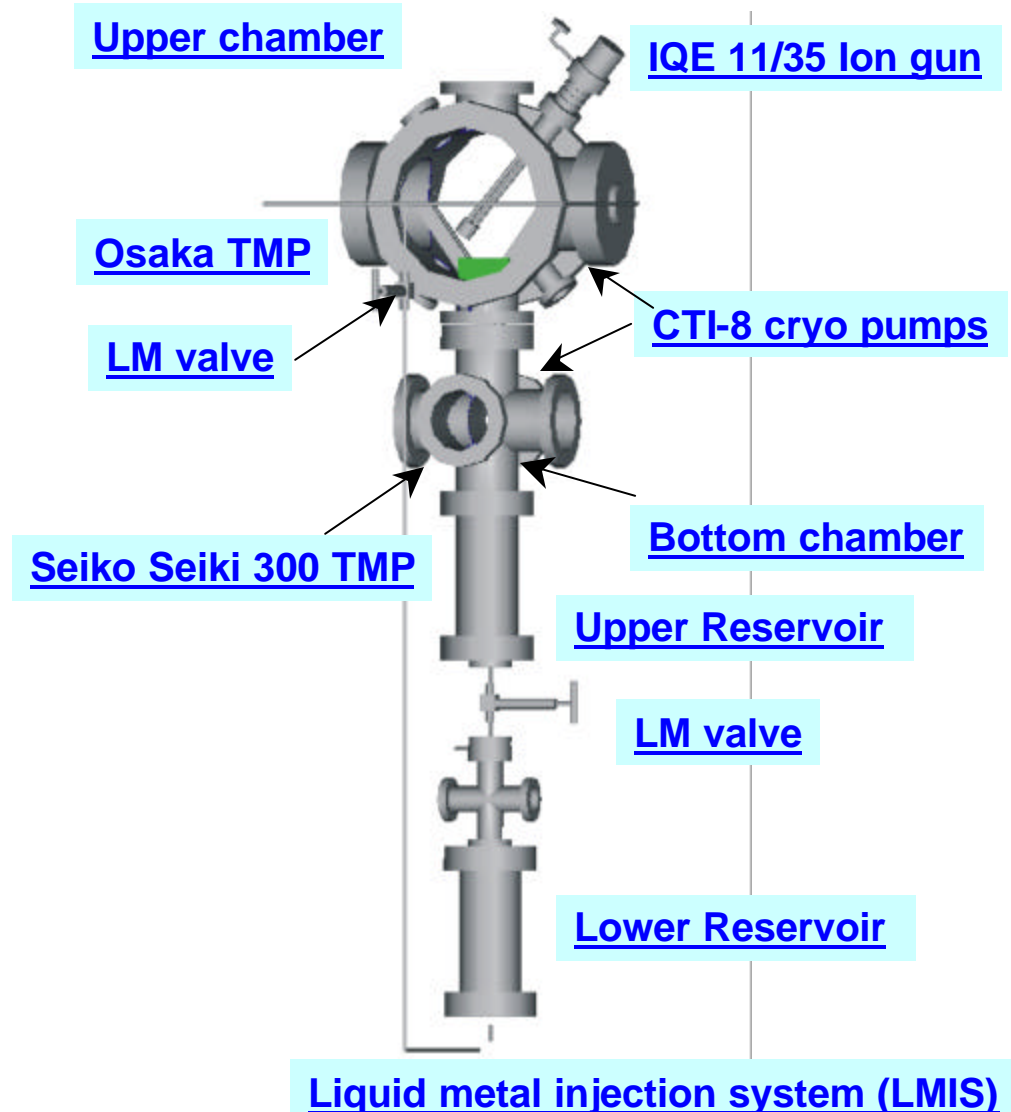
FLIRE assembly and construction



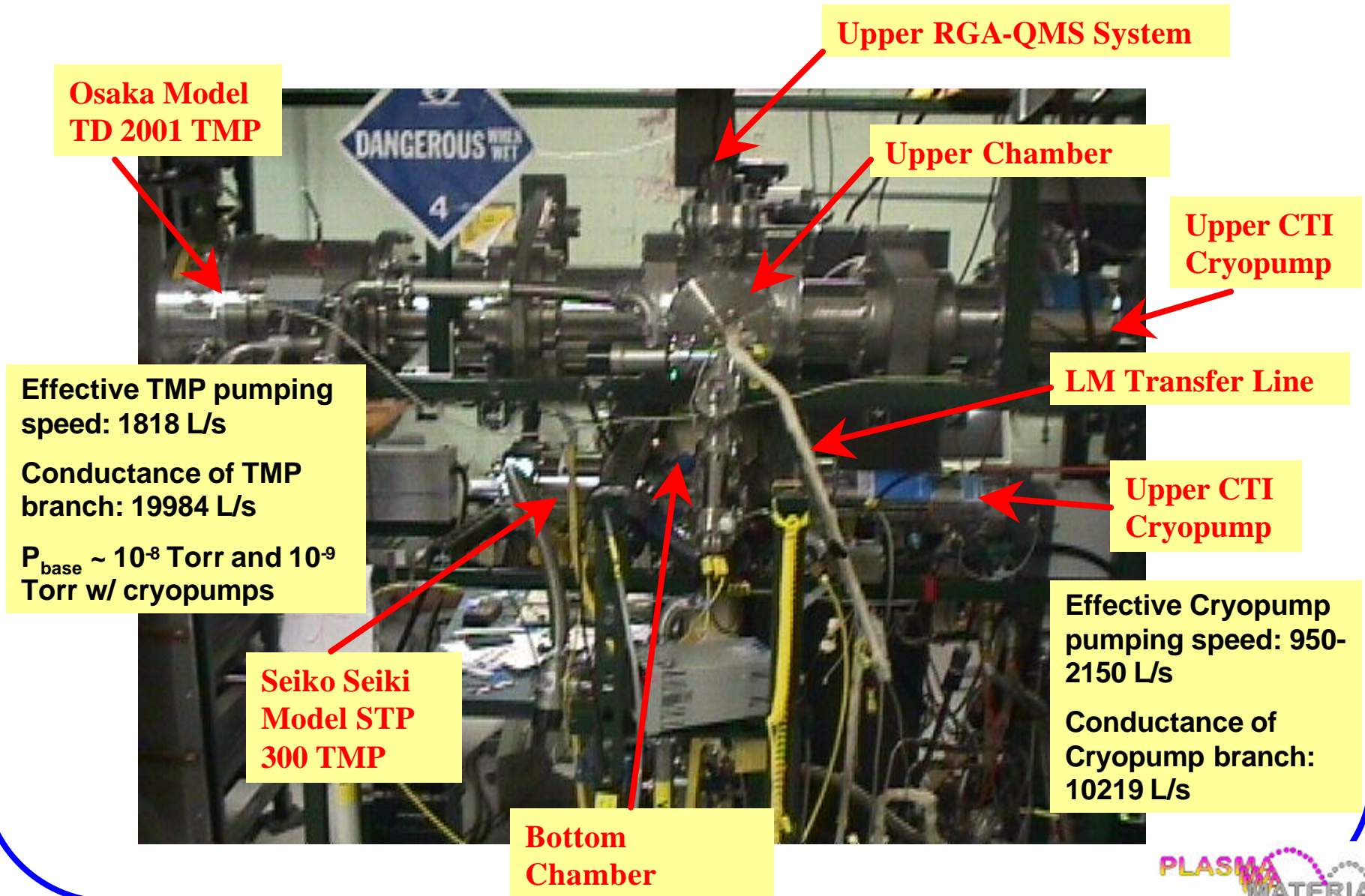
- All FLIRE components are installed and operational.
- Osaka Vacuum TMP Model TD 2001 is installed along with 2 CTI-8 cryopumps and Seiko Seiki Model STP 300 TMP.
- Liquid-metal injection system with liquid-metal compatible valves have been installed and tested.
- Ion gun and RGA-QMS systems have been tested.

General FLIRE Experimental Design

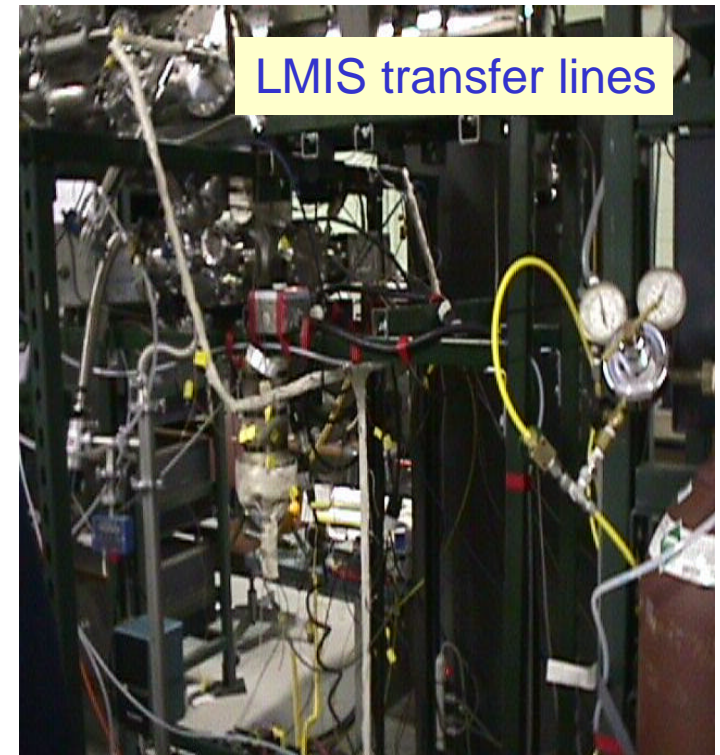
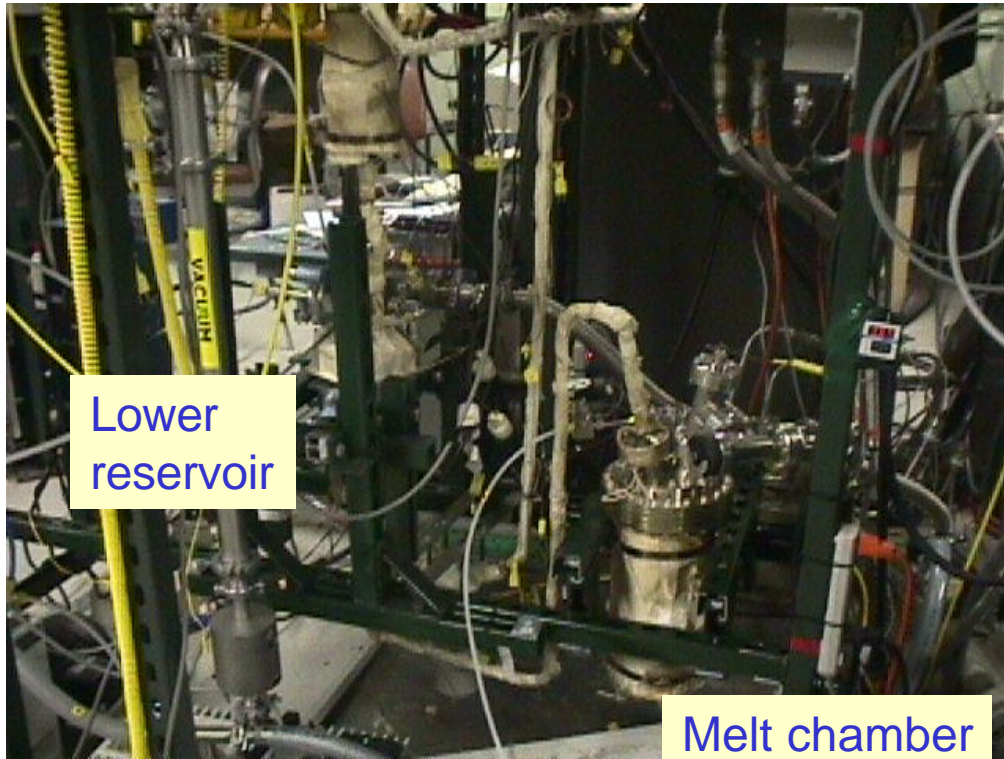
- The vacuum system is composed of 2 TMPs and 2 cryo pumps.
- SPECS IQE 11/35 Ion gun source.
- Upper and lower chamber.
- Reservoir and pressure tank
- RGA-QMS system for both chambers
- LM compatible valves



FLIRE vacuum system

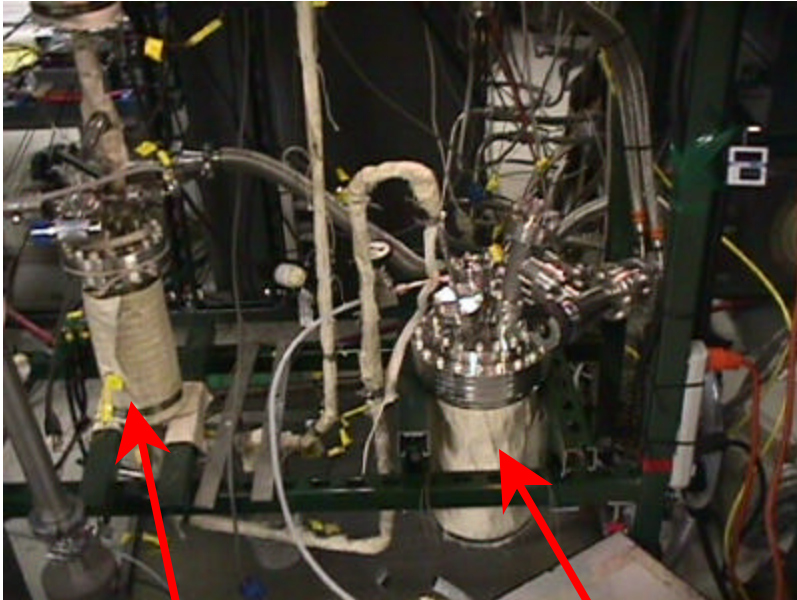


Liquid-Metal Injection System (LMIS)



- LMIS consists of an external melt chamber with transfer lines heated to ~ 300 C connected to lower reservoir shown on left.

Advantages of newly designed upper/lower reservoirs and external melt chamber

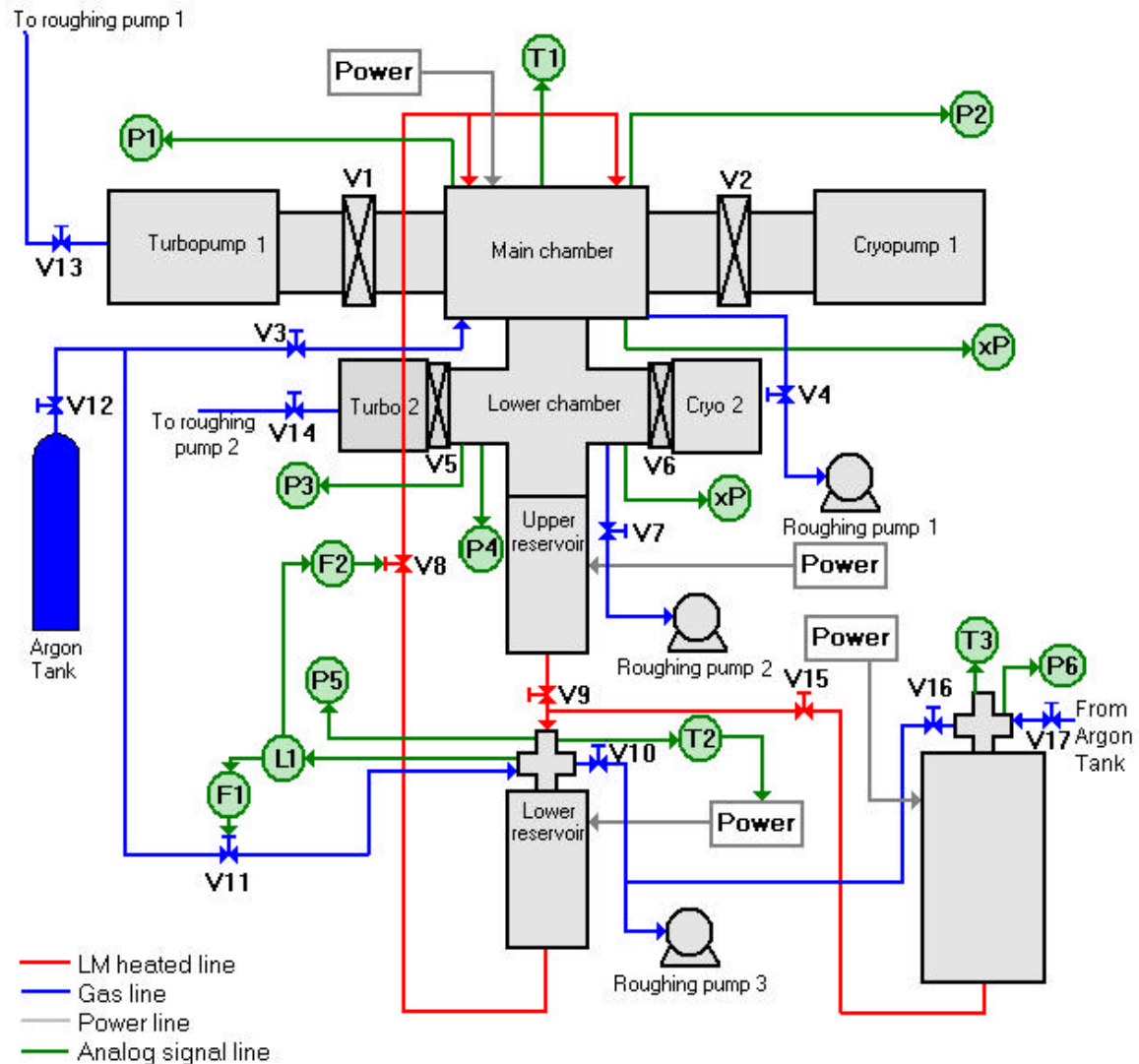


External melt chamber contains liquid Li. Liquid Li is then transferred to the lower reservoir.

- Liquid lithium can be transported from main FLIRE components to melt chamber in case of vent without compromising liquid Li
- Larger amounts of liquid Li can be accommodated
- Welded flanges on upper/lower reservoirs and melt chamber prevents any long-term reactions between liquid Li and Cu gaskets

General FLIRE operation

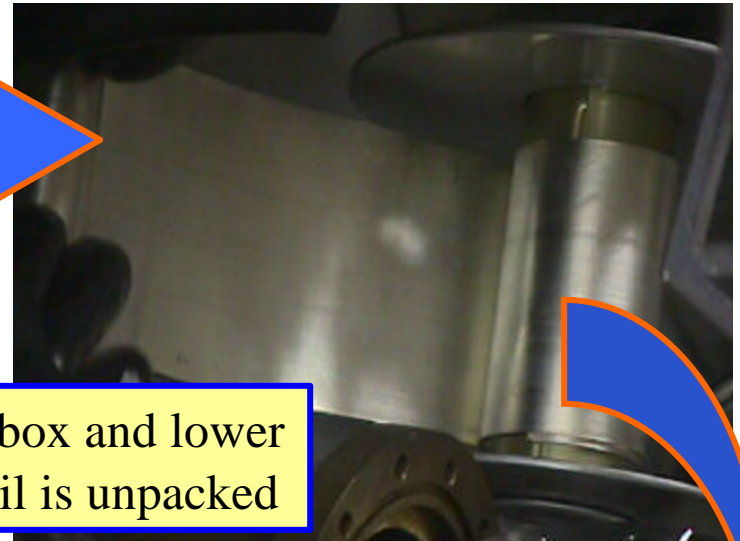
- Lithium loading procedure designed with use of external melt chamber and argon flow in a glove box
- Melting procedure melts lithium under argon atmosphere
- Once melting is in place, the liquid is transferred to the lower reservoir
- Then liquid Li is injected from the lower reservoir to the main chamber



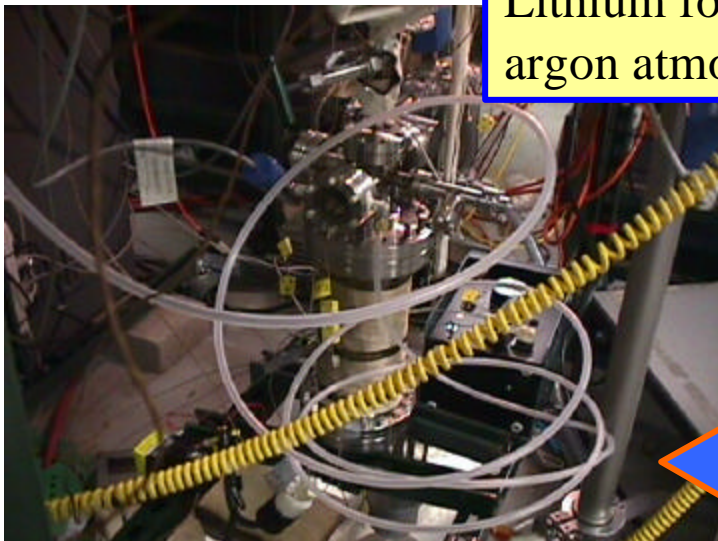
Lithium loading procedure in FLIRE



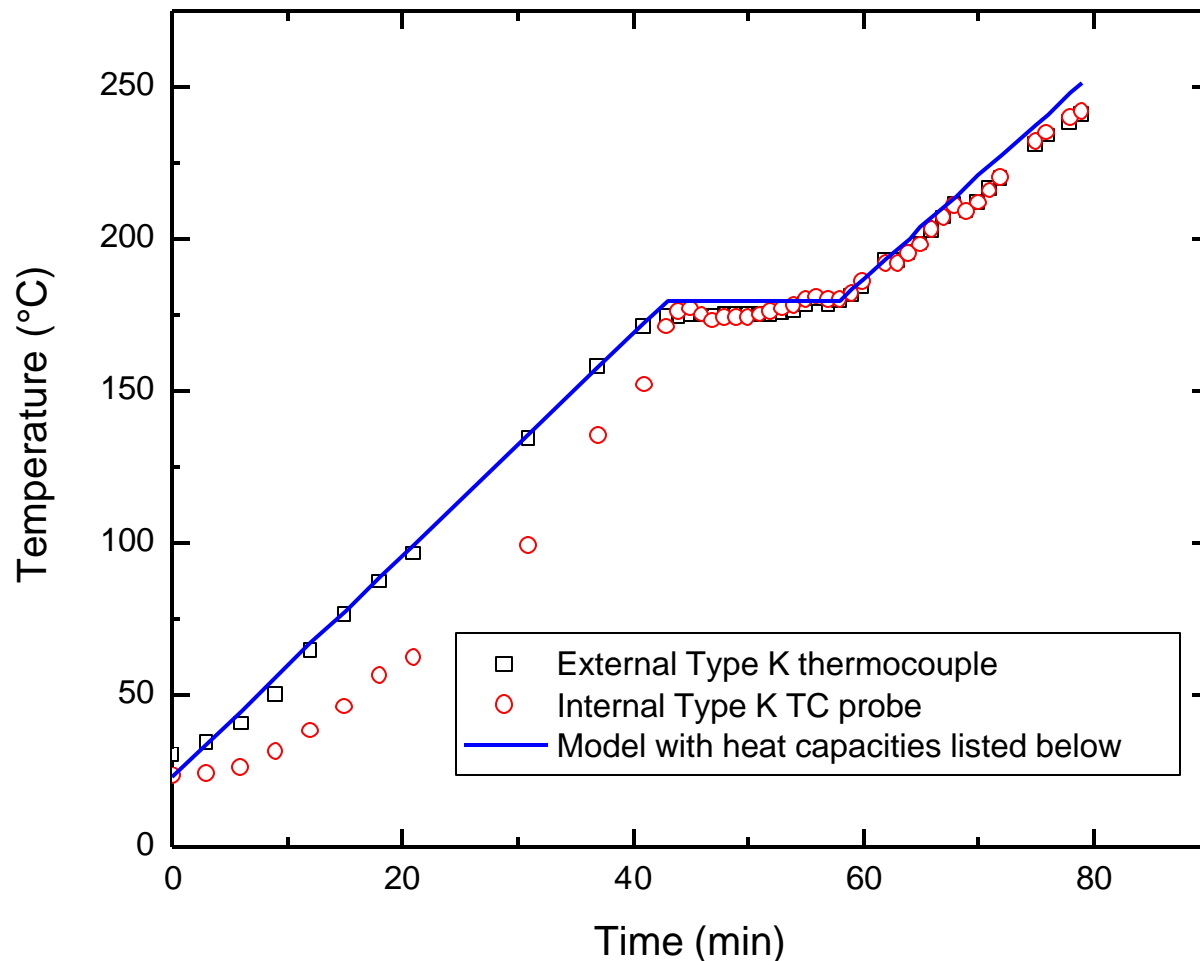
Argon flows in glove box and lower reservoir as lithium foil is unpacked



Lithium foil is inserted into the lower reservoir under argon atmosphere and then is installed on FLIRE



Lithium charge melting (cont'd)



$m = 1.5 \text{ kg}$

$P = 380 \text{ W}$

$C_{p,\text{sol}} = 4173 \text{ J kg}^{-1} \text{ K}^{-1}$

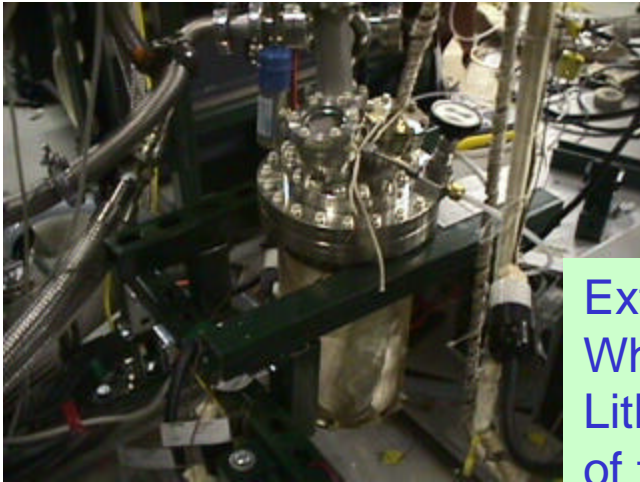
$C_{p,\text{liq}} = 4464 \text{ J kg}^{-1} \text{ K}^{-1}$

Lithium charge melting

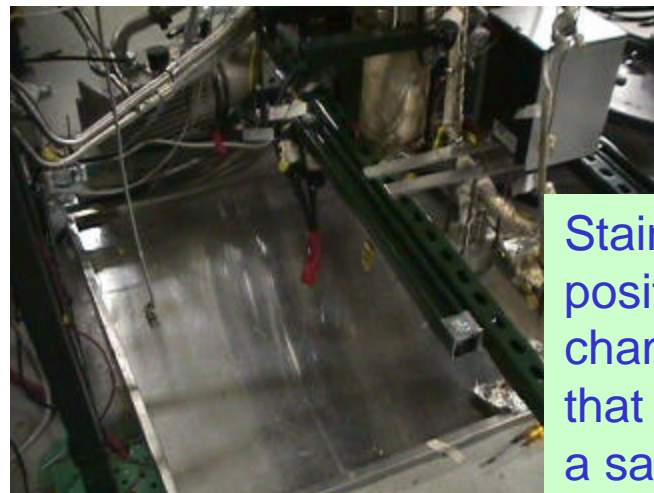
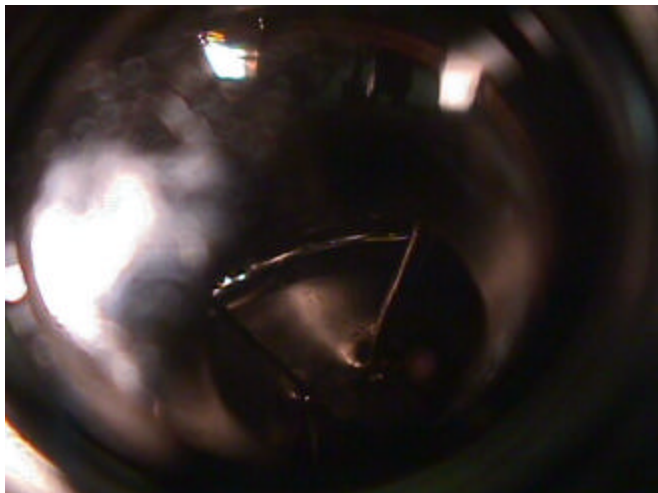
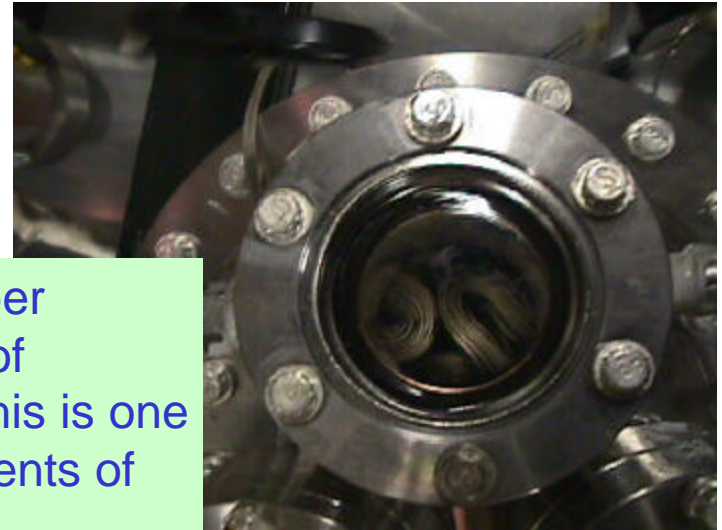
- A load of 1.5 kg of lithium was placed in the melting chamber (loads of up to ~ 3 kg of lithium can be accommodated)
- Thermocouples inside and outside the melt chamber monitored the temperature
- Model was done assuming constant heat capacities for the liquid and solid¹, and adjusting the transferred power value to 380 W (~25% of total power input)

1. T. B. Douglas, et al., J. Am. Chem. Soc., **77**(8), 2144-2150 (1955).

Liquid-Metal Injection System (LMIS)

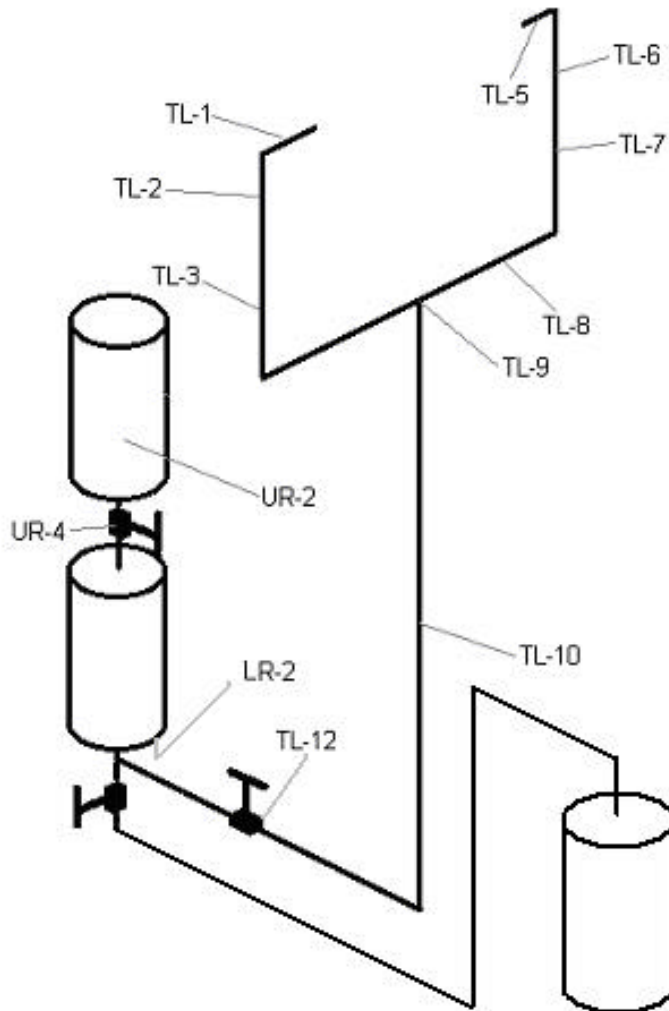


External Melt Chamber
Where about 1.5 kg of
Lithium is melted. This is one
of the major components of
the LMIS.



Stainless steel tray is
positioned under all
chambers and lines
that contain liquid Li as
a safety measure.

External heating system



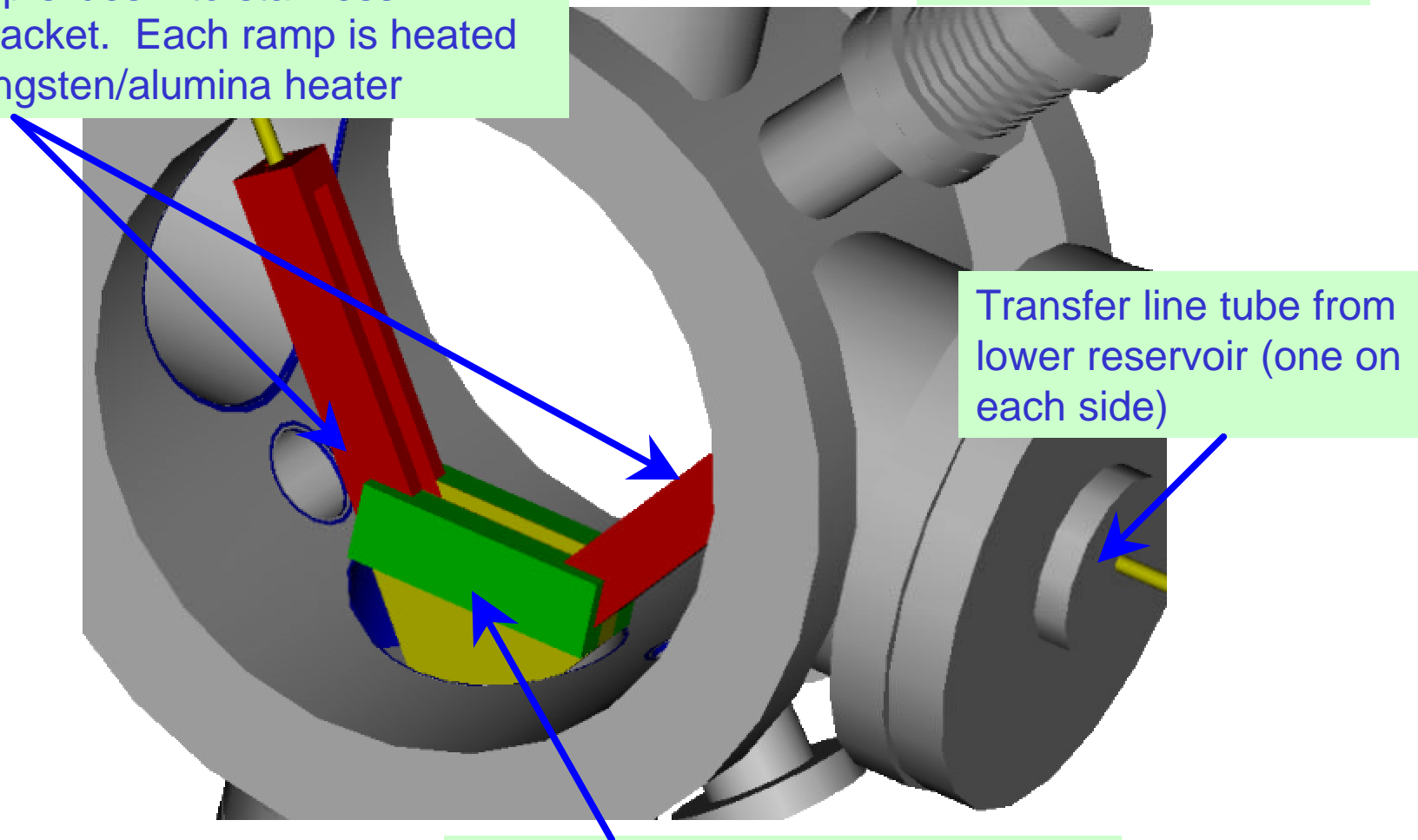
TC Id	Readout
TL1	243.6
TL2	227.2
TL3	236.8
TL5	233.1
TL6	270.8
TL7	271.6
TL8	238.6
TL9	210.5
TL10	333.5
TL12	185.1
UR2	304
UR4	197.7
LR2	300

- Heating tape wrapped around components
- All sections in contact with metal above MP
- Tubing and valves controlled with variable transformers
- Reservoirs controlled with PID controller

Liquid metal injection system ramps and bracket

SS ramp slides into stainless steel bracket. Each ramp is heated by a tungsten/alumina heater

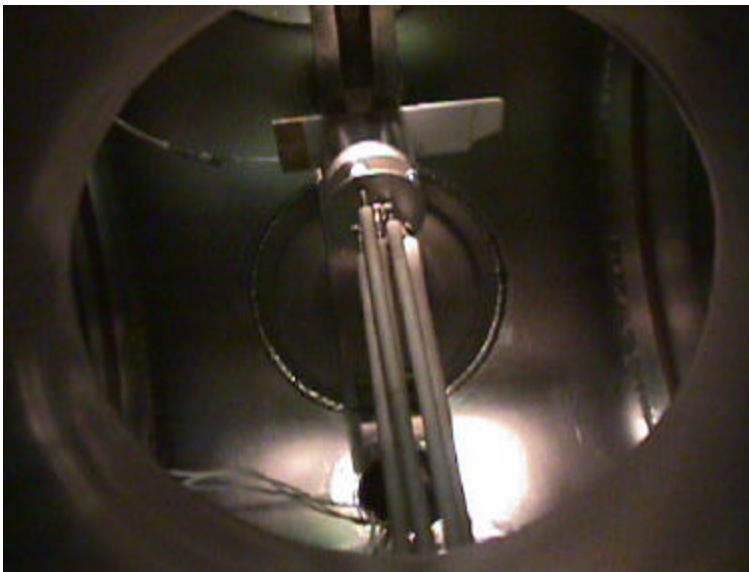
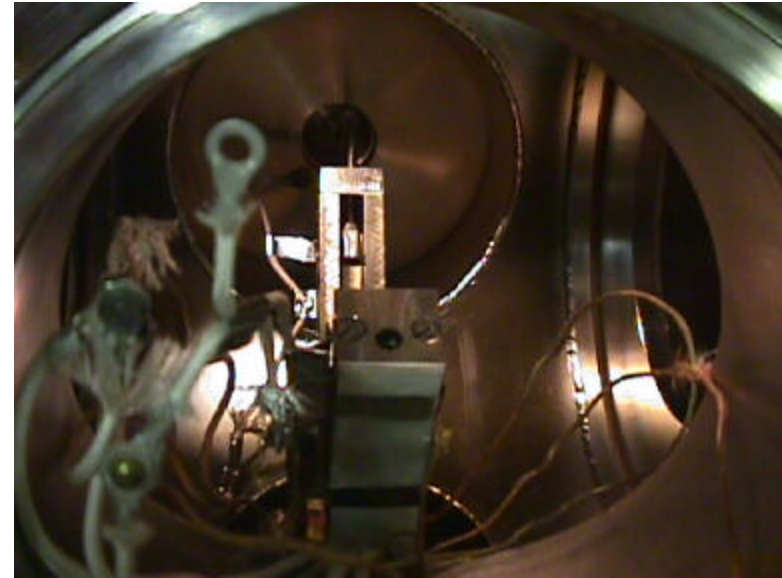
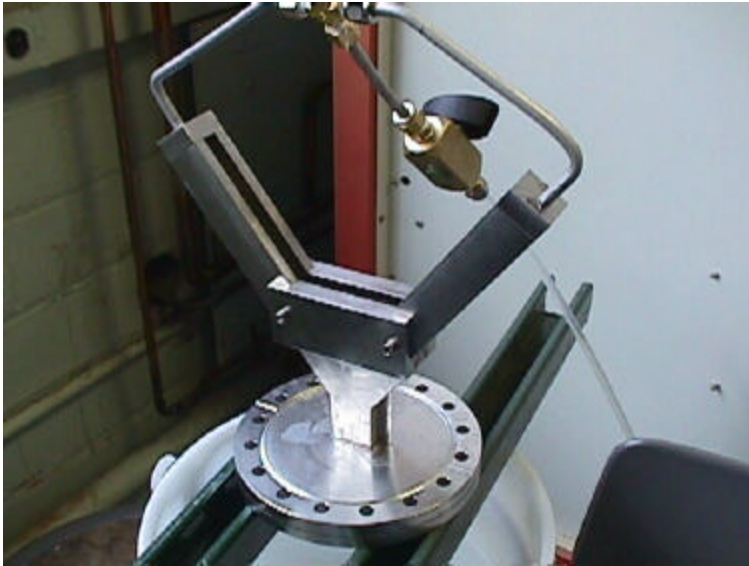
Ion gun flange location



Transfer line tube from lower reservoir (one on each side)

SS bracket is welded to a 6" flange attached to the upper chamber

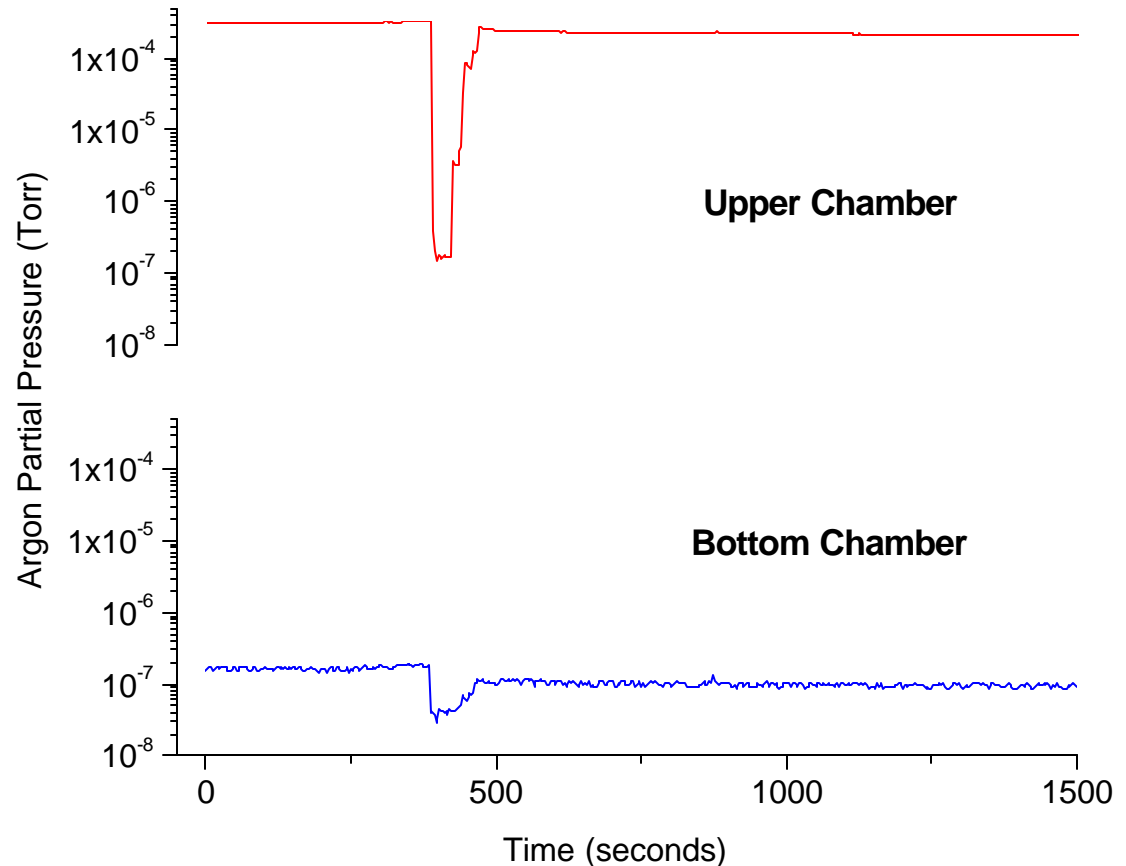
FLIRE ion gun/ramp system



- Dual ramp system installed
- Ion gun tested with small conducting dot
- Ion gun aligned to center of ramp channel

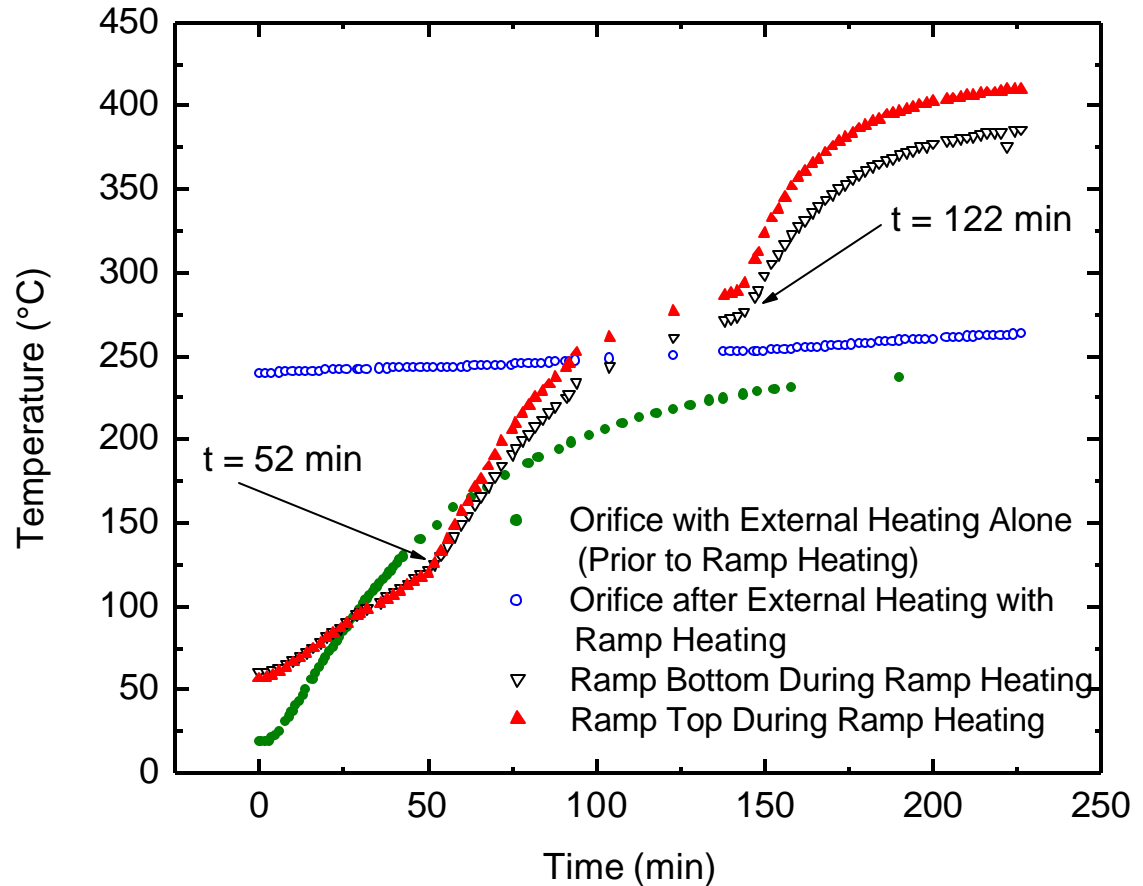
Upper and bottom chamber RGA argon traces

- Differential pumping provides over three orders of magnitude during Ar flow and one order of magnitude without flow
- 0.1 cm² orifice between upper and bottom chamber.
- Base pressure: 5×10^{-9} Torr
- With electron multiplier on, sensitivity increases by 100.

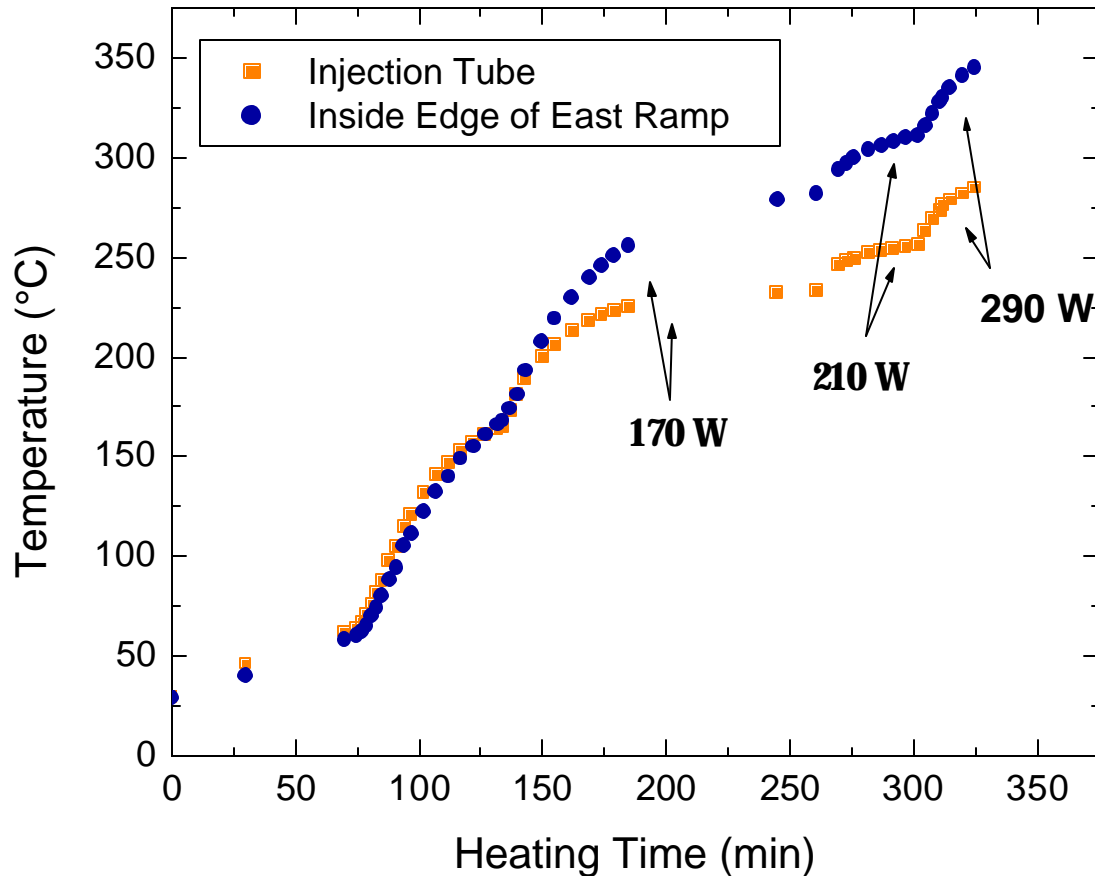


Internal Heating System tests

- 10-mil tungsten wire shaped to deliver uniform heating between 1/16 inch alumina plates
- Currents of 4 A are passed through wires to deliver 270 Watts of energy to the ramps raising the ramp temperatures as shown
- Orifice is heated externally by heating tape



Ramp Heating Profile

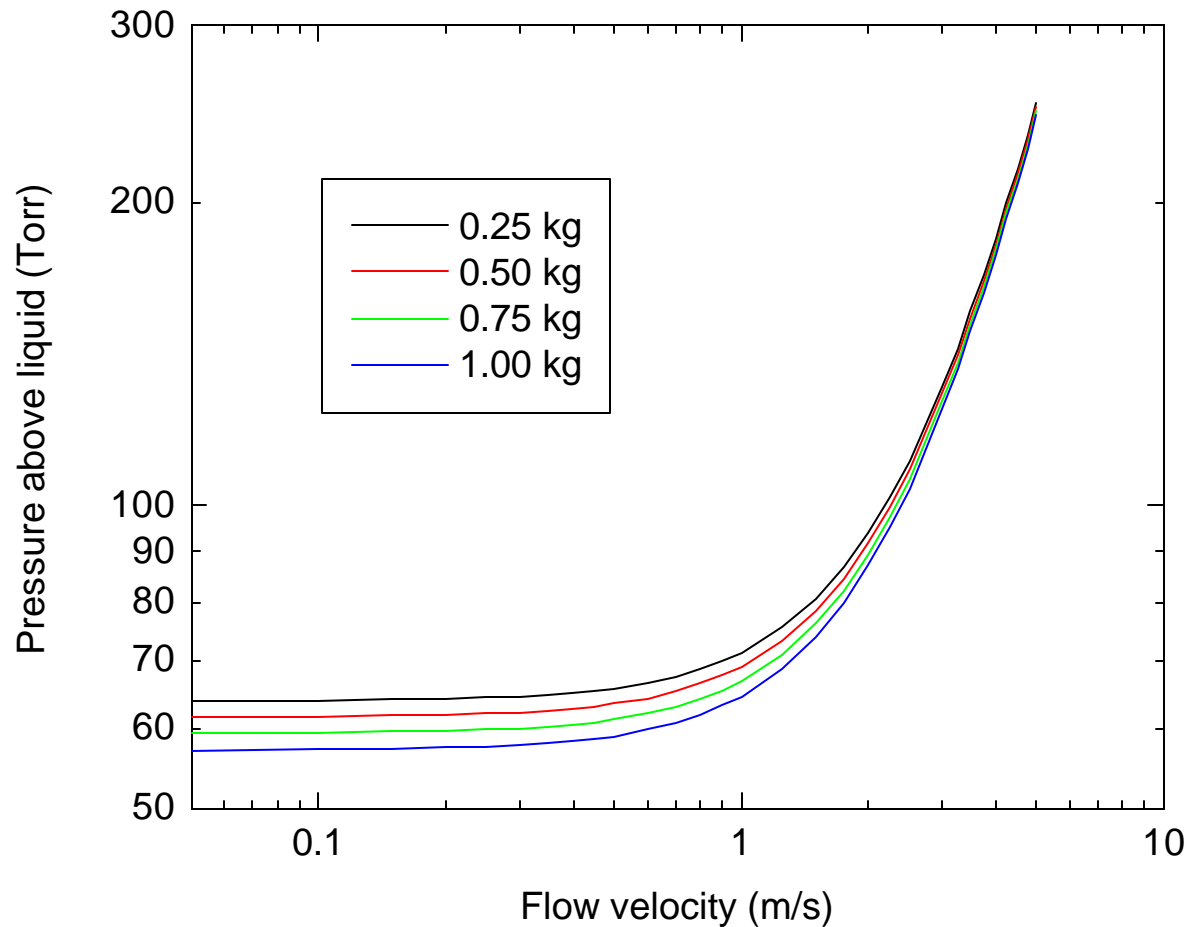


Each ramp heater power level has an upper temperature limit with each change in slope corresponding to an increase in power. The power levels of interest are indicated.

Liquid metal injection Tests

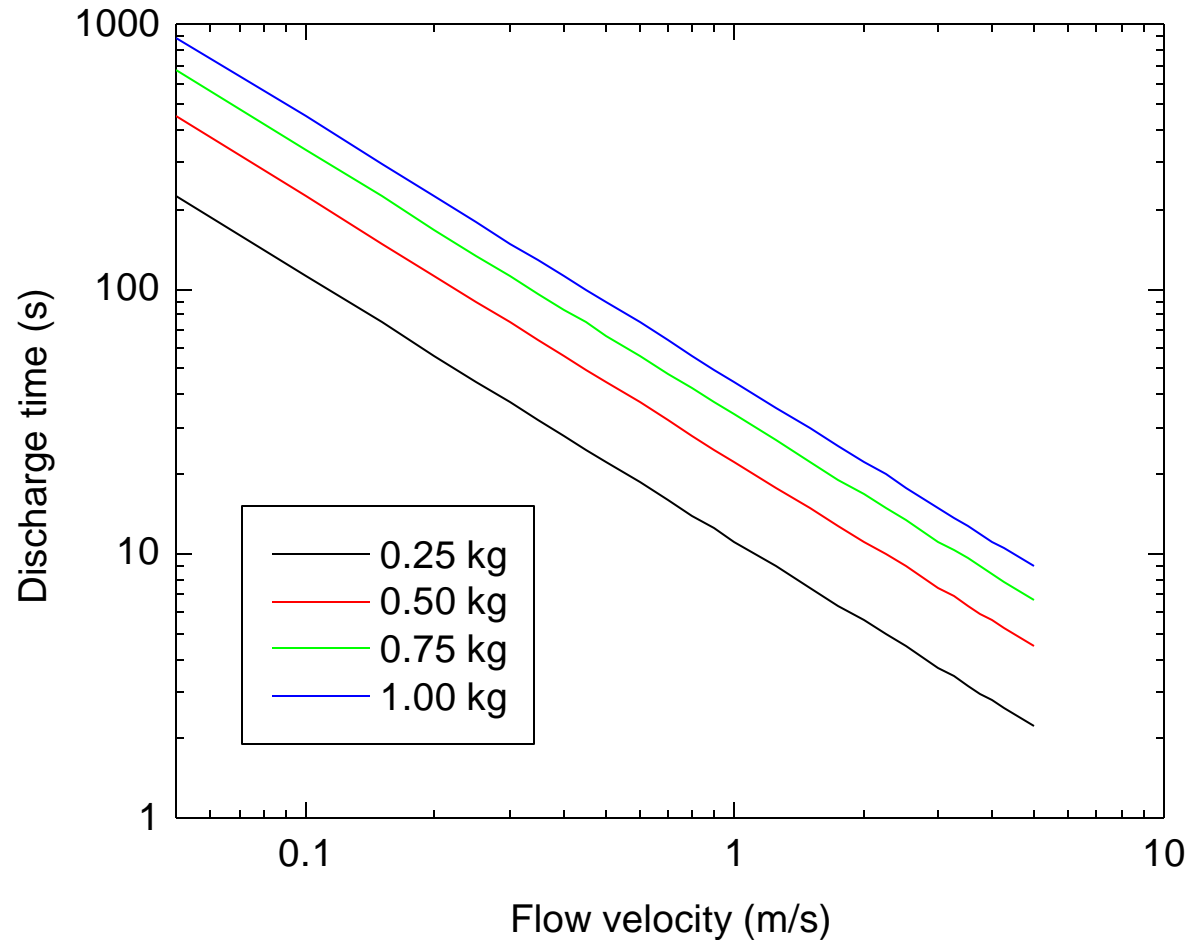
- Estimated pressure to lift the liquid metal from the lower reservoir to the chamber is ~ 60 Torr (ideal, no drops along the path).
- From experiment, LM starts to flow at 70 – 150 Torr. Some pressure drops at bends and splits in the line.
- Pressure needs to be increased as higher velocities are needed.
- Weak dependence of velocity with mass. Mass affects discharge time strongly.

Pressure vs liquid lithium flow velocity for various amounts of lithium



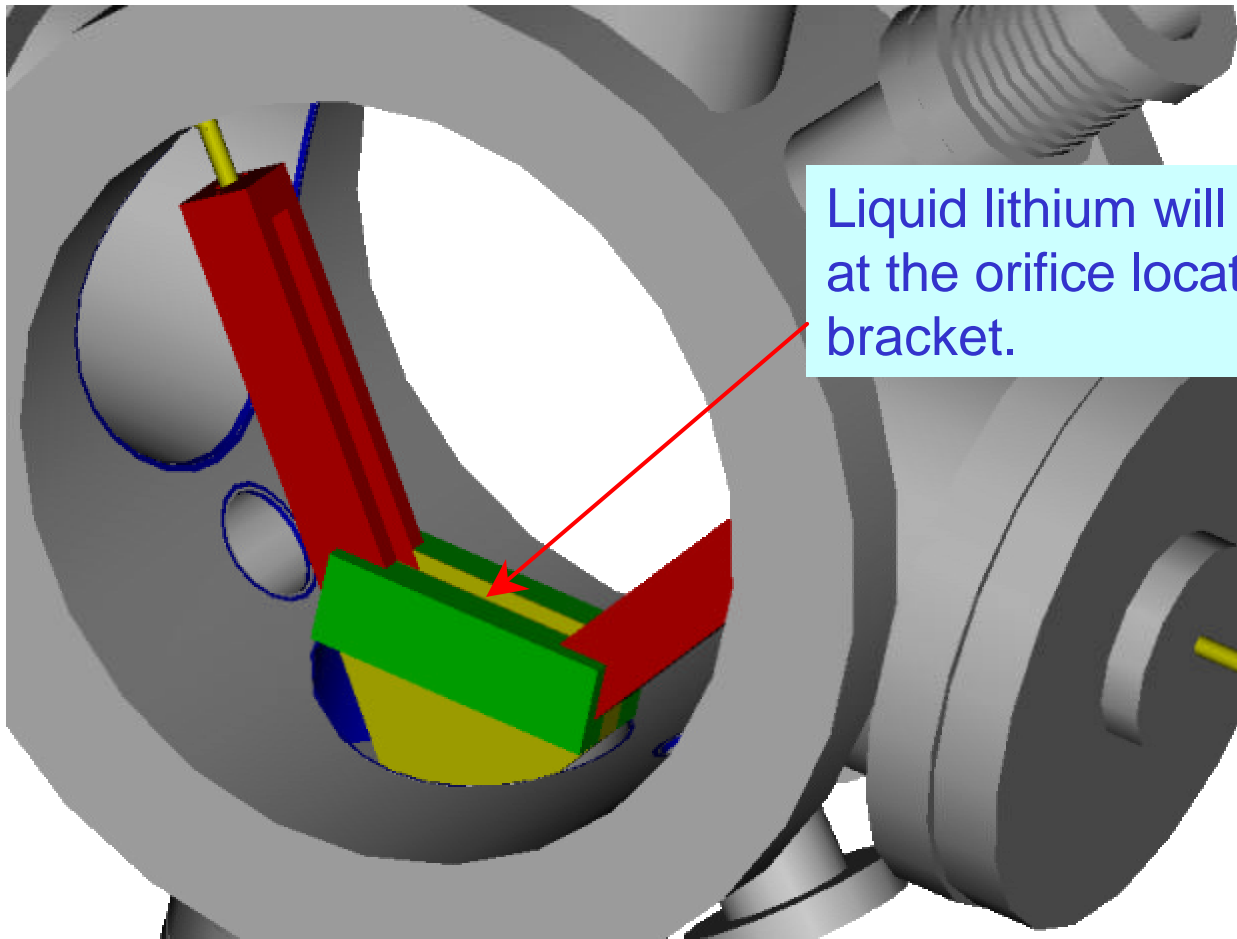
Pressure on liquid Li in bottom reservoir is raised when higher liquid lithium flow velocities are needed

Time of liquid lithium discharge vs liquid lithium flow velocity



Liquid lithium flow velocity also limits the time it takes for liquid metal to travel from lower reservoir chamber to the small orifice in the upper vacuum chamber

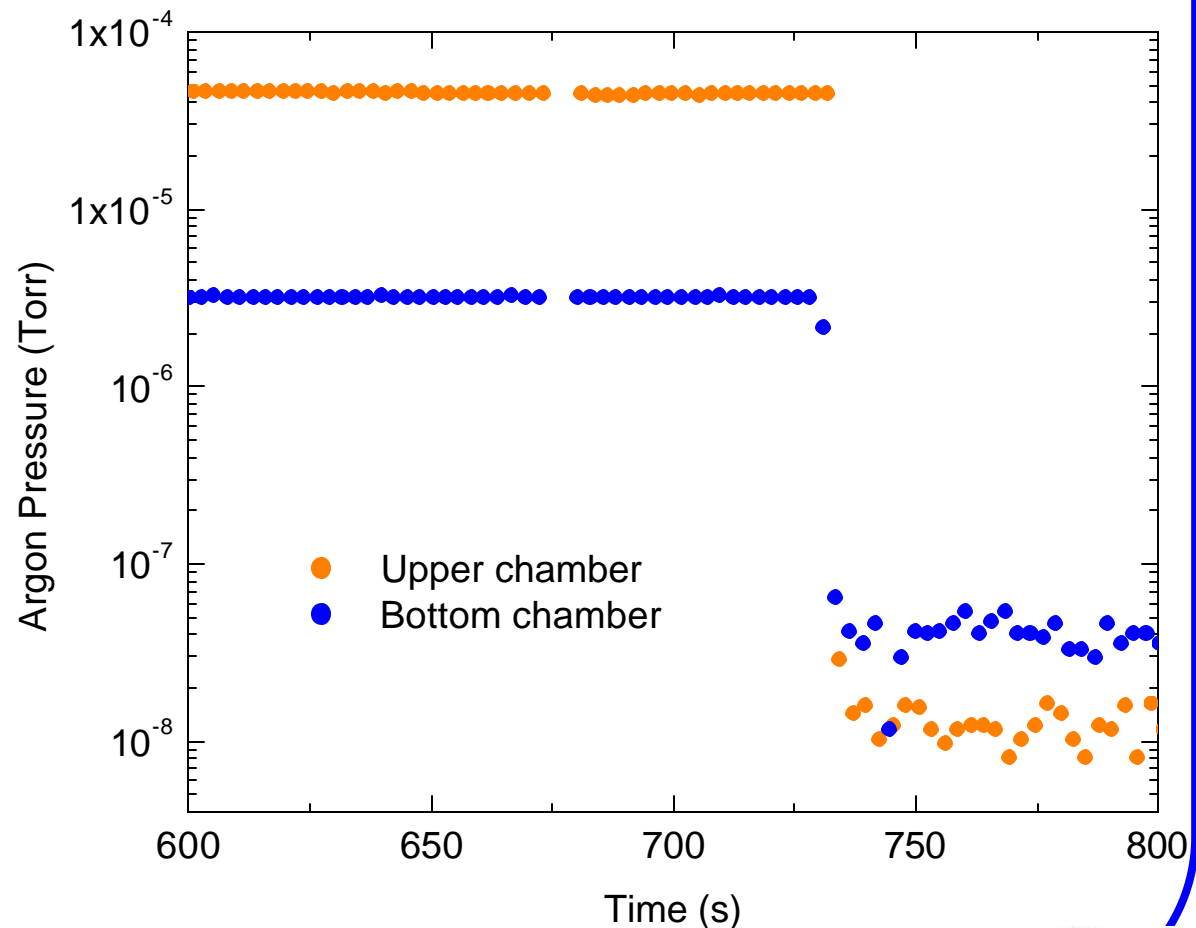
Liquid metal ramps



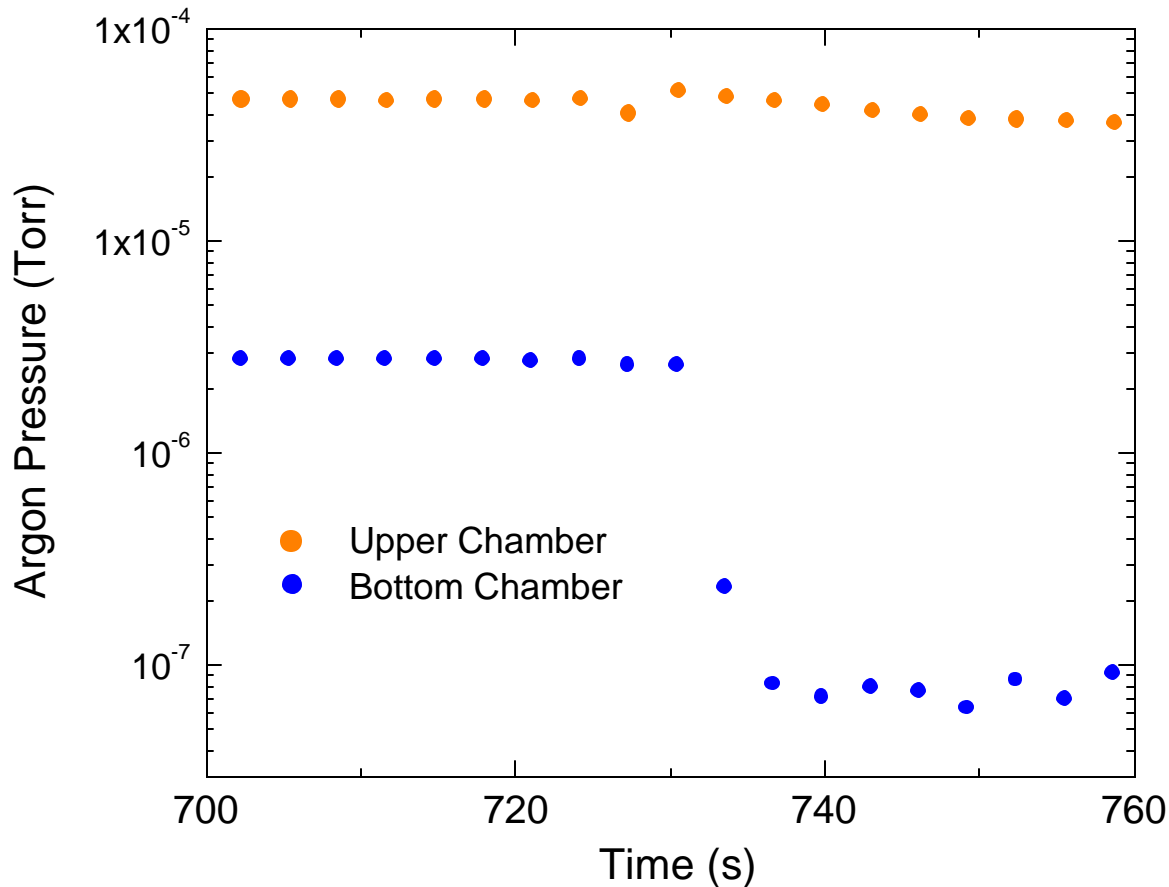
Liquid lithium will make seal at the orifice located down this bracket.

Partial pressure measurements in upper and bottom chambers

- Argon flows through an inlet on the top chamber. Bottom chamber trace with cryopump 20% open and TMP closed.
- Both chambers are separated by a 0.1 cm² orifice.
- Both argon partial pressures go down as the argon is shut off in the top chamber at about 730 seconds.



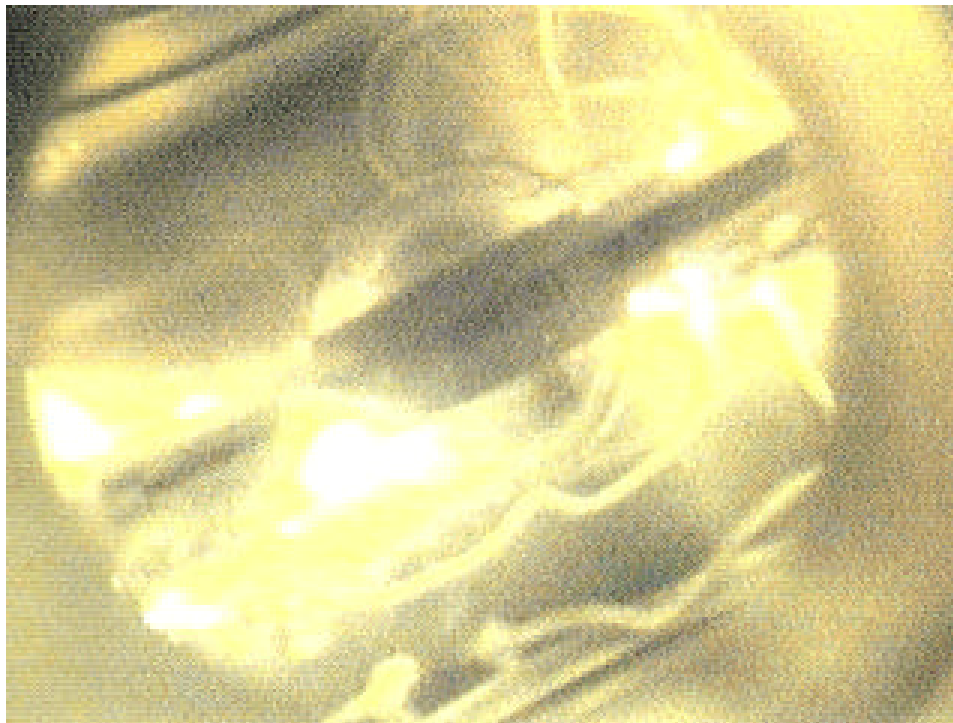
Evidence of Liquid Lithium Seal Formation



At ~ 750 s into the run, the liquid lithium shut the orifice cutting off the argon source to the bottom chamber.

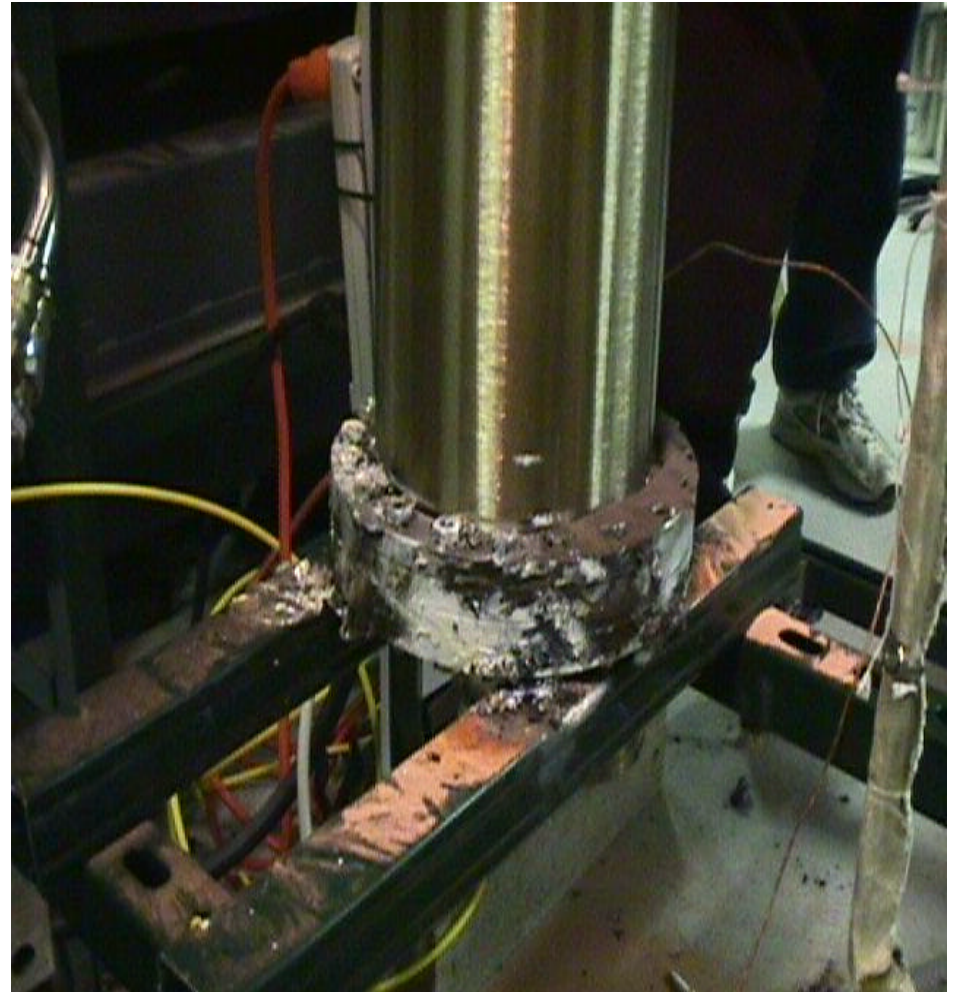
The size of the error bars are on the order of the size of the data point.

Flowing liquid lithium on SS ramps in FLIRE



Liquid lithium melt chamber spill

- Initially, 1.5 kg of Lithium was stored in the external melt chamber at 250° C in 175 Torr of Argon for 24 hrs.
- After prolonged storage at an elevated temperature, there was a reaction with the molten lithium and the copper conflat gasket.
- A lava like flow of droplets coming out of the seal between the flanges was observed to drip onto the stainless steel safety tray that is located under the FLIRE structure.
- These droplets were dull silver in color, indicating a reaction with oxygen in the atmosphere forming lithium oxide.
- Approximately 500 g of Lithium escaped the reservoir chamber.



Handling of Lithium Spill

- After noting Lithium leak, the melt chamber was immediately pumped down to depressurize it to stop the forceful ejection of molten lithium.
- Lithium flowed out of the chamber onto the safety tray. Depressurization of the melt chamber caused air to enter and force molten lithium all over the upper half of the reservoir chamber and into the roughing line.
- No lithium reached the roughing pump and there was no adverse reaction between lithium and roughing pump oil.
- Face masks were immediately put on and a class D Lithium Fire Extinguisher was immediately used to blanket the molten lithium around the melt chamber and on the safety tray, according to our safety procedure protocol.
- The safety tray effectively captured all of the escaping Lithium and there was no Lithium contact with any other potential material that would induce a hazardous reaction.



Analysis of Lithium Spill

- Safety procedure protocol was reviewed and fire suppression procedure deemed successful and more than adequate.
- Personal protective protocol was reviewed and breathing protection was upgraded.
- Analysis of stainless steel safety tray was assessed and deemed successful and adequate.
- Cause of event was determined to be a reaction between the molten Lithium, which was at an elevated temperature for an extended period of time, and the copper conflat gasket. As the Lithium reacted with the copper gasket, the gasket was consumed and degraded over time leading to the lithium spill.
- A chamber redesign was completed such that molten Lithium is no longer exposed to a copper conflat gasket for an extended period of time. The reservoir chamber now has a welded end on the bottom, which has been exposed to molten Lithium at 300°C for over two weeks without any reaction or event.
- Quick recovery of system (downtime ~ 2 weeks)



Safety protocol during lithium handling and cleaning



- **Inhalation Exposure Protection:** Face mask covering both the mouth and nose, Fans to establish a ventilation flow and remove dust/fumes.
- **Eye Exposure Protection:** Eye goggles that make a seal with the face around the eyes.
- **Skin Exposure Protection:** Neoprene gloves (.66 mm thick) to cover hands, but still allow finger mobility, Lab coats to cover exposed skin on the arms and neck, Long pants and covered shoes to protect legs and feet

Lithium Disposal



- Disposal of excess or used Lithium is done inside of a fume hood while wearing appropriate personal protective equipment.
- The Lithium is placed inside a Nalgene (HDPE) plastic bottle, which is then filled with Mineral Oil (Paraffin Oil, heavy) such that the Li is completely covered and no air is inside the container when sealed.
- The full containers are disposed of by the Division of Environmental Health and Safety, according to campus guidelines set by the University of Illinois, State of Illinois, and United States Government

Current system status

- Conducted lithium loading procedure under argon atmosphere
- Melted 1.5 kg of lithium in external melt chamber and transported liquid lithium to lower reservoir chamber
- Tested external heating systems
- Tested internal heating systems
- Tested liquid lithium injection and seal between upper and bottom chambers
- Improving liquid lithium injection techniques and testing for liquid lithium wetting of SS ramps

FLIRE data analysis

- Partial pressure of injected ions in bottom chamber gives diffusion coefficient
- Can vary injection point and flow speed to verify data reduction model
- Can measure hydride formation through thermal desorption measurements

Particle profile for the model

- General solution for density profile, $n(x,t)$:

$$n(x,t) = \int_0^\infty f(\mathbf{x}) G_2(x, \mathbf{x}, t) d\mathbf{x}$$

- Green's function of the second kind:

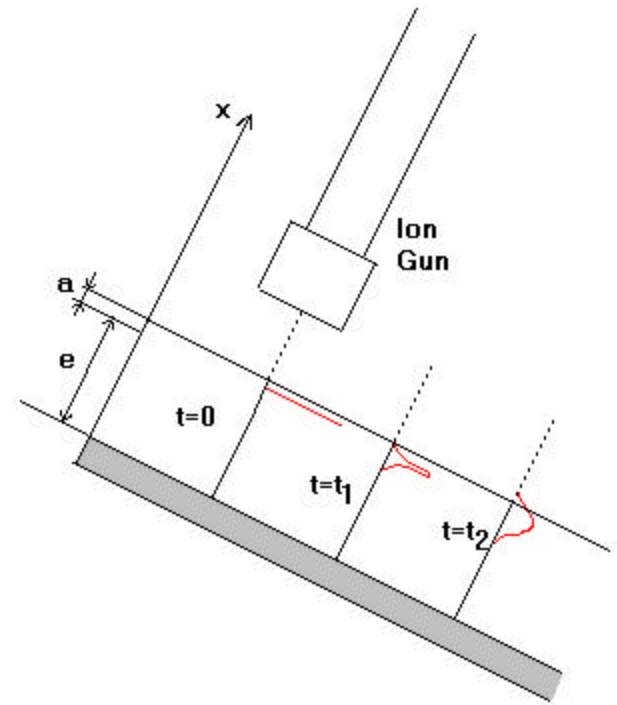
$$G_2(x, \mathbf{x}, t) = \frac{1}{\sqrt{(4\mathbf{p})Dt}} \left\{ e^{-\frac{(x-\mathbf{x})^2}{4Dt}} + e^{-\frac{(x+\mathbf{x})^2}{4Dt}} \right\}$$

- Initial particle profile, a and σ from TRIM

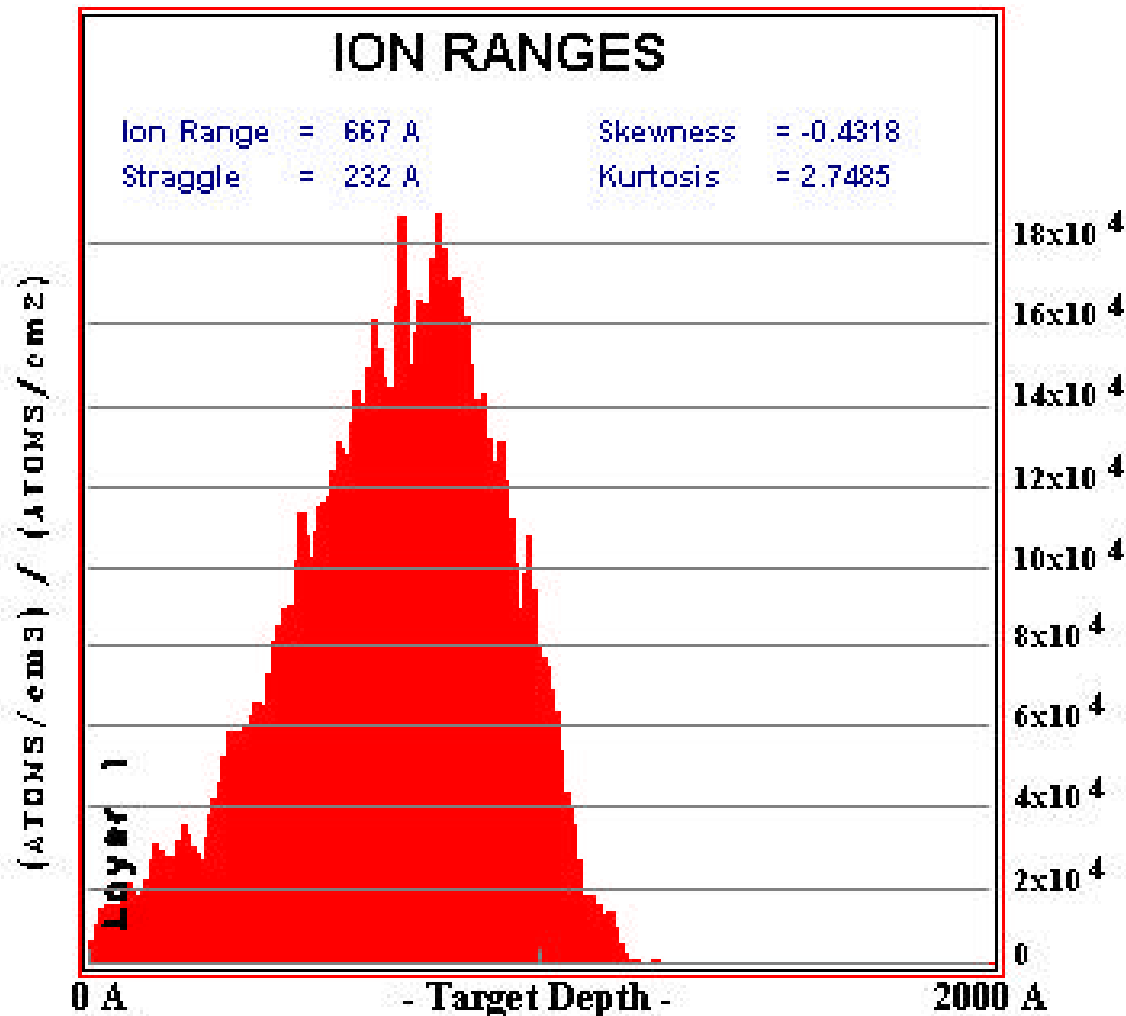
$$f(x) = \frac{1}{\sqrt{(2\mathbf{p})\mathbf{s}}} e^{-\frac{(x-e)^2}{2\mathbf{s}^2}}$$

- Particles retained in flowing liquid, $r(t)$,

$$r(t) = \int_0^{e+a} n(x,t) dx$$



Implanted particle profile for 800 eV H in Li target



RGA Data interpretation

- The release rate into the second chamber is related to the RGA equilibrium pressure measurement by:

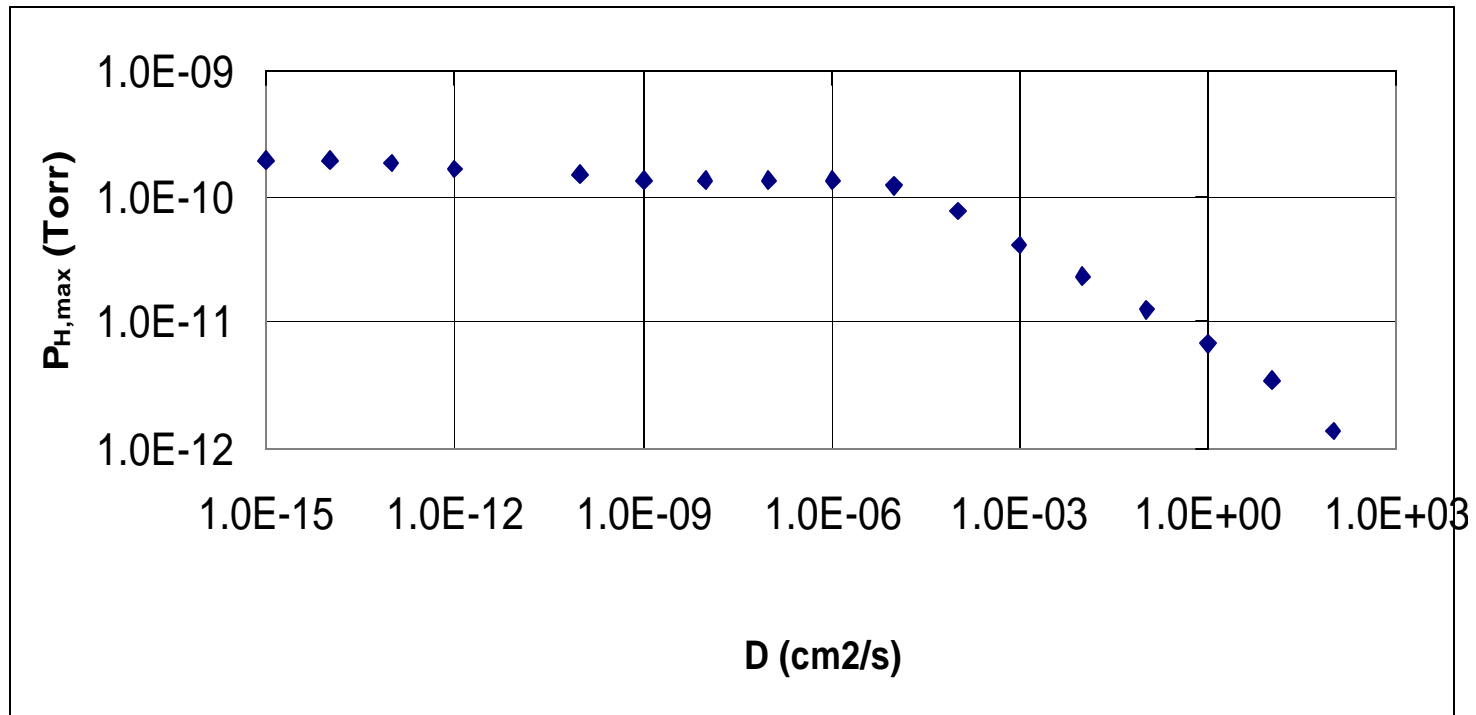
$$P = \frac{r(t_2)kT}{S}$$

- The effective pumping speed, S , was determined from pump down curves on the actual chamber to be $7 \times 10^{-7} \text{ m}^3/\text{s}$
- t_2 is the time it takes for the liquid lithium to travel from the ion gun strike point to the second chamber

Parameters for the model

- A flow velocity of 1 m/s and a travel distance of 0.1 m in the ramp gives a transit time from the striking point to the second chamber entrance, t_2 , of 0.1 s.
- The thickness of the film e is assumed to be 1 mm
- The penetration depth a , and the standard deviation s for the initial condition $f(x)$ are given by the TRIM code, and are a function of energy and incidence angle. For 1 keV hydrogen in lithium, $m = 1000$ Å and $s = 500$ Å. For 250 eV hydrogen in lithium, $m = 250$ Å and $s = 110$ Å
- The diffusion coefficient, D , can be calculated as a function of the amount of particles released in the second chamber, $r(t)$

Relationship between P_H and D



$P_{H,max}$ as a function of D . All peak pressures are measurable by the available RGA system for a D range between 10^{-6} to 10^{-3} cm²/sec.

FLIRE plans and upgrades

- Continue liquid lithium injection until reliable and repeatable technique is achieved.
- Retention measurements of H^+ and He^+ particles in liquid lithium under a variety of conditions.
- Upgrade FLIRE to a **functional prototype**
 - DC plasma discharge source (first part of Phase II)
 - Magnetic field coils and permanent magnets could be added to study MHD and entry effects
 - Electrodes added to simulate eddy currents

Conclusions

- LMIS operation
- Safety tests complete and lithium handling procedures in place
- Pressure requirements for liq Li flow rate
- New LMIS design and tests
- Liquid Li injection tests
- He and H retention tests to be conducted in the near future
- Plasma source to be added (STTR Phase II)
- Could add magnetic fields and electrodes to simulate MHD effects

Acknowledgements

- G. H. Miley, NPL Associates, Inc (STTR funding Phase I) DE-FG02-01ER86134
- ALPS/DOE funding
- Undergraduate students: Andy Simnick, Dan Rokusek, Sarfraz Taj, Gabe Burt and Jason Tillery.